COLUMBIUM ALLOY EXTRUSION PROGRAM

PHASE V: TUBING PROGRAM
INTERIM REPORT VII
15 OCTOBER 1962 - 15 JANUARY 1963

BASIC INDUSTRIES BRANCH
MANUFACTURING TECHNOLOGY LABORATORY
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WRIGHT-PATTERSON AIR FORCE BASE, OHIO

ASD PROJECT NO. 7-775

The development of a tube blank extrusion process for B-66 (Cb-5Mo-5V-1Zr) and X-110 (Cb-10W-1Zr-.1C) is described. The tube reducing program for the extruded blanks is discussed.

(Prepared under Contract AF33(600)-40700 by E. I. du Pont de Nemours and Company, Inc., Baltimore, Maryland, James S. Clark)

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FOREWORD

This Interim Technical Documentary Progress Report covers the work performed under Contract AF33(600)-40700 from 15 October 1962 to 15 January 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with E. I. du Pont de Nemours & Company, Inc., Baltimore, Maryland was initiated under Manufacturing Methods Project 7-775, "Columbium and Columbium Alloy Extrusion Program". It is being accomplished under the technical direction of Mr. T. S. Felker of the Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. James S. Clark, Development Engineer, Metals Center, Baltimore, is the engineer directly responsible for the work. Others who cooperated in the development program were: Dr. A. W. Dana, Jr., Technical Supervisor, Mr. R. W. Felber, Operations Supervisor, and Mr. J. A. Crane, Laboratory Engineer.

Wolverine Tube Division of Calumet Hecla, Inc., is the subcontractor to E. I. du Pont de Nemours & Company, Inc., for the tube reduction development program. Mr. J. C. Huber is the engineer directly responsible for the work at Wolverine. Mr. F. C. Eddens, Manager, Special Metals Department, and Mr. L. B. Moorman, Project Metallurgist, also contributed to this program.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

PUBLICATION REVIEW

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INTERIM TECHNICAL PROGRESS REPORT NO. 7-775 (VII)
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COLUMBIUM AND COLUMBIUM ALLOY EXTRUSION PROGRAM
TUBING PROGRAM

ABSTRACT

The development of an extrusion process for B-66 (Cb-5Mo-5V-1Zr) and X-110 (Cb-10W-1Zr-.1C) tube blanks was continued in the second three-month period of Phase V. The outside surfaces of the extruded tube blanks were satisfactory; the inside surfaces were improved but still contained defects. The effects of process parameters are described. Conditioned tube blanks were given the first tube reducing pass. The X-110 blanks reduced satisfactorily but the B-66 blanks cracked radially. The tube reducing program is discussed.

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I. INTRODUCTION

This report summarizes the results of work performed in the second three-month period of Phase V of the "Columbium Alloy Extrusion Program". The preceding report, Interim Report VI (1)[1] covered the initial three-month period of Phase V, July 15, 1962 - October 15, 1962. In that report was described the revision of Phase V of the original contract from the pilot production extrusion of a "T" shape of alloy D-31 (Cb-10Ti-10Mo) to a development program on tubing of the alloys B-66 (Cb-5Mo-5V-1Zr) and X-110 (Cb-10W-1Zr-.1C). The reproducibility of the D-31 "T" extrusion process developed in the contract was demonstrated in Phase IV, thus obviating the need for a pilot production run (Phase V). The "T" extrusion development program, which encompassed the first four phases of the contract, is described in reports (2), (3), (4), (5), and (6).

The goal of Phase V is to produce three sizes of tubing of the columbium alloys B-66 and X-110 as follows:

1/2" O.D. x .062" wall (40% by weight)
3/8" O.D. x .062" wall (40% by weight)
1/4" O.D. x .018" wall (20% by weight)

The processing steps are -

1) double vacuum arc melt 8" diameter ingots
2) extrude to 4" rounds

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[1] Numbers in parentheses refer to the Bibliography at the end of the report.

3) machine hollow billets and re-extrude to tube blanks

4) tube reduce to finish tubing

Wolverine Tube Company, Allen Park, Michigan, is subcontractor to Du Pont for the tube reduction program. Wolverine and Du Pont have engaged in a cooperative program to determine a satisfactory reduction-anneal schedule for the B-66 and X-110 tubes.

II. SUMMARY OF PROGRESS

An additional B-66 ingot and an X-110 ingot, each 8" diameter x approximately 135 pounds, were melted and converted to 4" round billet stock by warm extrusion. This brought the total of 8" diameter ingots used in this phase of the contract to six, three of each alloy. The additional ingots were required to offset the low yield incurred in the initial tube blank extrusion campaign.

The major effort in this three-month period has been the development of the tube blank extrusion operation. A process was developed which produced tube blanks with much improved surfaces over the initial extrusions in the program. The improvement has been attributed to the refinement of the glass lubrication practice used in conjunction with a cone die. Transverse checks persist on the inside surface, however.

A total of twenty-three extrusions were attempted, all at temperatures in the range of 3000-3200°F. Die type, glass lubrication practice, and billet design were the major parameters investigated. Seventeen of the twenty-three billets were extruded but only thirteen tube blanks were considered satisfactory for the tube reduction program.

Considerable difficulty was encountered in conditioning. A major problem arose in conditioning the outside surface of the tube blanks. Repeated straightening attempts at ambient temperature resulted in fracture. A deep pickling operation combined with hand-grinding to remove surface defects was the most effective method of conditioning.

Wolverine Tube has completed the first tube reduction on the initial batch of four X-110 tube blanks. The two B-66 tube blanks cracked radially in the first tube reducing pass. A possible source of the cracks is residual extrusion surface defects, present despite extensive conditioning prior to the tube reduction.

III. TECHNICAL DATA

A. ADDITIONAL MATERIAL

An additional 200 pounds of B-66 and 200 pounds of X-110 starting materials were added to the program. An 8" diameter ingot of each alloy was prepared by double vacuum arc-melting at the Du Pont Metals Center. The additional ingots were required to replace material which was considered unsalvageable from the initial tube blank extrusions. The ingots were melted and extruded to 4" round billet stock by the same method described in the previous report (1) for the original four ingots in the program.

The breakdown extrusion details for the two steel-canned ingots appear in Table 1. The extrusions were satisfactory, but a 2-1/2" nose burst occurred on the B-66 extrusion. The extrusions were sectioned into billet stock 6-7" long.

TABLE 1

BREAKDOWN EXTRUSION OF 8" DIAMETER B-66 AND X-110 INGOTS

Ingot	Extruded Dia., in.	Extrusion Ratio	Temperature °F[1]	Extrusion Pressure, ksi Upset	Extrusion Pressure, ksi Running	K,[2] ksi	Appearance
X-110-346	4.24	3.6:1	2040	83	87	65	Good
B-66-347	4.6	3.0:1	2000	102	93	93	2-1/2" nose burst; good surface

1 Corrected optical pyrometer reading on steel-canned ingot.

2 Extrusion constant calculated using upset pressure.

B. TUBE BLANK EXTRUSIONS

Development of an extrusion process to produce B-66 and X-110 tube blanks with defect-free surfaces, good dimensional control, and of sound quality is a major objective of the contract. The tube blanks produced in the first extrusion campaign of the initial three-month period (1) had adequate dimensional control but fair-to-poor inside and outside surfaces. The defects were attributed to improper glass lubrication practice. Two extrusion campaigns were conducted at the Du Pont Metals Center in this reporting period. Emphasis was placed on the development of a glass practice, coupled with the proper type die, which would eliminate surface defects.

The general procedure for the tube blank extrusions consisted of the following:

1. Heat billet in induction heater under protective atmosphere, at rate of 150-200°F/minute. Measure temperature by optical pyrometer. Soak 5 minutes.
2. Transfer billet from heater to press in 35 seconds or less. Roll heated billet over powdered glass on glassing table in transfer; apply powdered glass to bore manually.
3. Place selected glass pad against modified shear type die only.
4. Lubricate liner with mixture of MoS_2 and "Oildag".[1]
5. Insert die and mandrel in press after heating in 750°F. oven for approximately 1/2 hour.

[1] Proprietary lubricant manufactured by Acheson Colloids Company.

6. Place two 1" thick graphite pads heated to 2200°F. directly behind billet. Place a copper follower block 2" thick behind graphite.
7. Extrude at ram speeds up to 5"/second.

Two sizes of tube blanks were recommended by Wolverine Tube for starting stock for tube reduction, as follows:

1) 1.750" O.D. x .30" wall (80%)
2) 1.750" O.D. x .25" wall (20%)

Item 1) will be the starting stock for the 1/2" and 3/8" O.D. tube sizes, and Item 2) the starting stock for the 1/4" O.D. tubing.

The dies and mandrels for tube blank extrusion were described in detail in the Interim Report VI (1). Briefly, both a 90° included angle cone type die and a modified shear type die were used. The working surfaces of the dies and mandrels were zirconium-oxide coated; the ground coating was approximately .030" thick. The extrusion size would permit approximately 0.050" "cleanup" on both the inside and outside diameters to the tube blank sizes given above, Items 1) and 2).

The details of the two extrusion campaigns are listed in Table 2 as "Second Series" and "Third Series", the "First Series" having taken place in the initial reporting period. The extrusions are listed in the order performed. The principal parameters investigated were -

1) billet design
2) billet temperature
3) die type
4) glass lubrication practice

TABLE 2 BLANK

B-66 AND X-110 TUBE Pressu

Billet[1]	Billet Design	Die Type	Extrusion Ratio	Billet Temp., °F.	Extrusion B.T.	Pressu Ru
"SECOND SERIES":						- est
X-110-278-01	Fig. 1-A	cone	7.5:1[2]	3090	172	106
B-66-279-06	Fig. 1-C	cone		3130	N.A.	119
X-110-278-03	Fig. 1-A	flat		3380	137	111
X-110-280-03	Fig. 1-A	flat		3330	156	125
B-66-277-04	Fig. 1-A	flat		3210	154	
B-66-277-01	Fig. 1-A	flat	8.4:1[3]	3160	156	
"THIRD SERIES":						144 157
X-110-278-01	Fig. 1-A	cone	7.5:1	3030	180	-
B-66-347-03	Fig. 1-C	cone		3060	185	1
X-110-346-02	Fig. 1-C	cone		3010	190	-
X-110-346-01	Fig. 1-A	cone	8.4:1	3050	186	157
B-66-347-02	Fig. 1-A	cone	7.5:1	3030	190	1
B-66-347-01	Fig. 1-A	cone	8.4:1	3160	190	152
X-110-280-07	Fig. 1-A	cone	8.4:1	3170	190	-
B-66-347-04	Fig. 1-A	cone	7.5:1	3200	189	130
B-66-279-04	Fig. 1-A	cone		3210	190	144
X-110-346-03	Fig. 1-A	cone		3080	169	153
B-66-277-05	Fig. 1-A	cone		3100	190	131
B-66-279-05	Fig. 1-A	cone		3100	189	137
X-110-278-02	Fig. 1-A	cone		3030	168	-
X-110-280-01	Fig. 1-B	flat		2930	172	1
B-66-277-03	Fig. 1-B	flat		2970	190	-
B-66-279-02	Fig. 1-B	cone		3110	190	
X-110-278-04	Fig. 1-B	flat		2960	190	

1. listed in order of extrusion

2. die opening: 1.80" O.D. x 1.125" I.D.

3. die opening: 1.80" O.D. x 1.225" I.D.

TABLE 2 BLANK EXTRUSIONS

B-66 AND X-110 TUBE

on	Billet Temp., ^{o}F.	Pressure, ksi Extrusion B.T.	Running	Results
2	3090	172	--	Stalled
	3130	N.A.	est. 137	Extremely poor; deep tears
	3380	137	106 → 120	I.D., O.D. defects
	3330	156	119 → 129	I.D., O.D. defects
	3210	154	111 → 124	Very poor; holes through wall
3	3160	156	125 → 144	Light defects, best of series
	3030	180	144 → 156	O.D. good, I.D. poor
	3060	185	157 → 177	O.D. score from die wash; I.D. poor
	3010	190	--	Stalled
	3050	186	154	O.D. good, I.D. fair
	3030	190	--	Stalled
	3160	190	157 → 181	Light O.D. defects, I.D. fair
	3170	190	190	O.D. good, I.D. fair
	3200	189	152 → 172	Light O.D. defects, I.D. fair
	3210	190	--	Stalled
	3080	169	130 → 148	O.D. good, I.D. fair
	3100	190	144 → 162	O.D. good, I.D. fair
	3100	189	153 → 168	Light O.D. defects, I.D. fair
	3030	168	131 → 151	O.D. good, I.D. fair
	2930	172	137 → 141	Poorest O.D. of series, I.D. fair
	2970	190	--	Stalled
	3110	190	190	O.D. good, I.D. fair
	2960	190	--	Stalled

2

How these parameters were varied and what effect they had on extrusion quality are described below:

1. Billet Design

The basic billet design is shown in Figure 1-A. The 90° included-angle cone nose completely filled the cavity of the cone die prior to upset.

The two other billet designs were the 60° included-angle cone nose, Figure 1-B, and the flat nose with 1/4" 45° chamfer, Figure 1-C. The I.D. of the billets in Figure 2 - 1.225" - was used for the approximate 80% of the extrusions of the nominal size of 1.75" O.D. x 0.3" wall. For the remaining 20% of the extrusion at the nominal size of 1.75" O.D. x .25" wall (larger mandrel required) the billet I.D. was increased to 1.325". For both cases the nominal mandrel-cold billet I.D. clearance was 0.100" (diametral). Further, the amount of diametral clearance for certain billets was reduced from 0.100" to 0.050" to determine whether this clearance would influence the extruded inside surface. No obvious effect was observed.

The basic billet design with the 90° included-angle cone nose, Figure 1-A, proved to be the most satisfactory when used with a cone type die. The billet design with the sharper nose, Figure 1-B, resulted in excessive chilling at the extreme nose end. Two of the four billets of this design stalled the press after upset, Table 2. The flat nose billet design with 1/4"-45° chamfer, Figure 1-C, proved unsatisfactory. Severe die

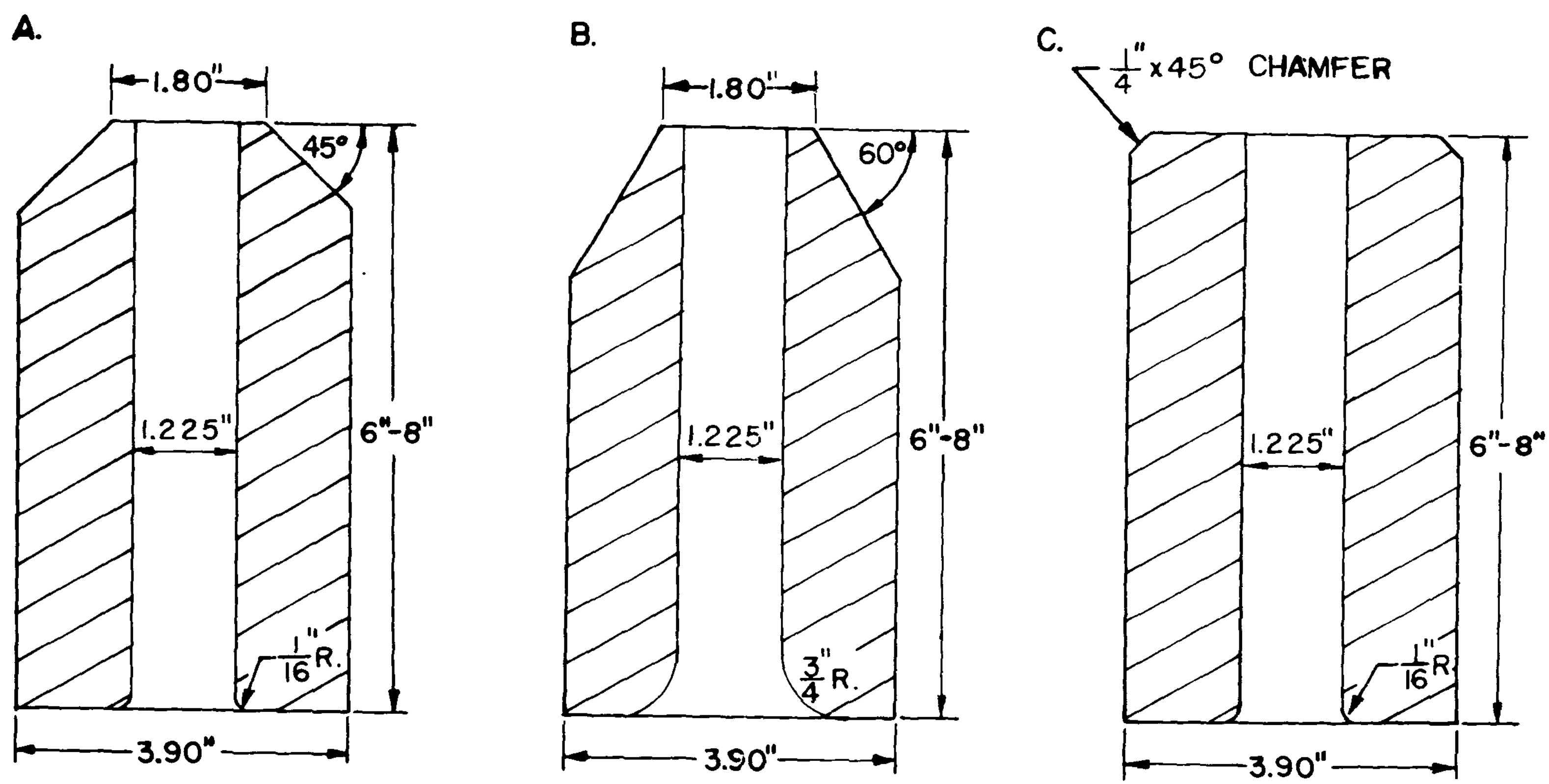


FIGURE 1

THREE BILLET DESIGNS FOR TUBE BLANK EXTRUSION

wash occurred when one billet was extruded through a cone die. The flat entry of the billet into the die cavity apparently ruptured the ZrO_2 coating on the die face, leading to die wash. This conclusion is based on earlier "T" extrusion work (6) which showed that use of a pure columbium nose plug (relatively soft) on the D-31 (Cb-10Ti-10Mo) billets eliminated die coating failure.

2. Billet Temperature

Billet temperatures for the two extrusion series described in this report were generally in the range of 3000-3200°F. This range is approximately 200°F. lower than the temperature range for the initial tube blank extrusion series reported earlier (1). This reduction was achieved due to the more effective glass lubrication practice developed in this stage of the program. The billet temperature used for each extrusion was the estimated minimum temperature which would result in a complete extrusion, the factors of glass practice and die type being considered. Similar temperatures were selected for the B-66 and X-110 extrusions.

3. Die Type

Two die types - a 90° included-angle cone and a modified shear with 1/2" entry radius - were investigated. In the "Second Series", Table 2, four of the six extrusions were made through flat dies. In the first extrusion of this series, in which a cone die was used, the billet was upset but failed to extrude. Examination

of the billet revealed a prominent circumferential lip at the location of the die seal to the liner. The cause of the lip was traced to a damaged liner seal. Therefore, this campaign was abbreviated. Modified shear dies were used on all extrusions except one. The glass pads placed against the flat die effectively covered the damaged seal. In general the surfaces produced by the flat dies were poor. Since this problem is associated with the glass lubrication practice it will be discussed in the next section.

In the "Third Series", Table 2, the final series in this period, the emphasis was placed on the use of cone dies. The results of the prior tube blank extrusion campaigns indicated that smoother surfaces could be obtained with cone rather than flat dies.

4. Glass Lubrication Practice

The "Second Series" of tube blank extrusions, Table 2, employed mainly modified shear dies. Glass pads of different composition were placed against the die to form a cone of glass during extrusion. In addition, glass powder was applied 1) manually to the bore of the heated billet and 2) to the outside surface by rolling the billet down a glassing table in transfer from the heater to the press. Each of the three points of glass application - against the die, on the barrel, and in the bore of the billet - was treated as a separate function. Different pad compositions were tried to determine what glass(es) would form a satisfactory cone

under the conditions of extrusion speed and billet temperature employed. The shape of this cone during extrusion of the billet obviously cannot be determined unless the extrusion is purposely stopped. However, the shape of the copper follower, which is only partially extruded, presented an indication of the glass cone formed against the flat die. A pad approximately 4" O.D. x 1-1/4" I.D. x 1-1/2" thick, made up of layers of a very high melting glass cloth, was considered satisfactory in extruding the tube blanks from about 3200°F, and 1-3"/sec. ram speed.

At the outset of the tube blank extrusions, two general premises were held regarding glass practice, as follows:

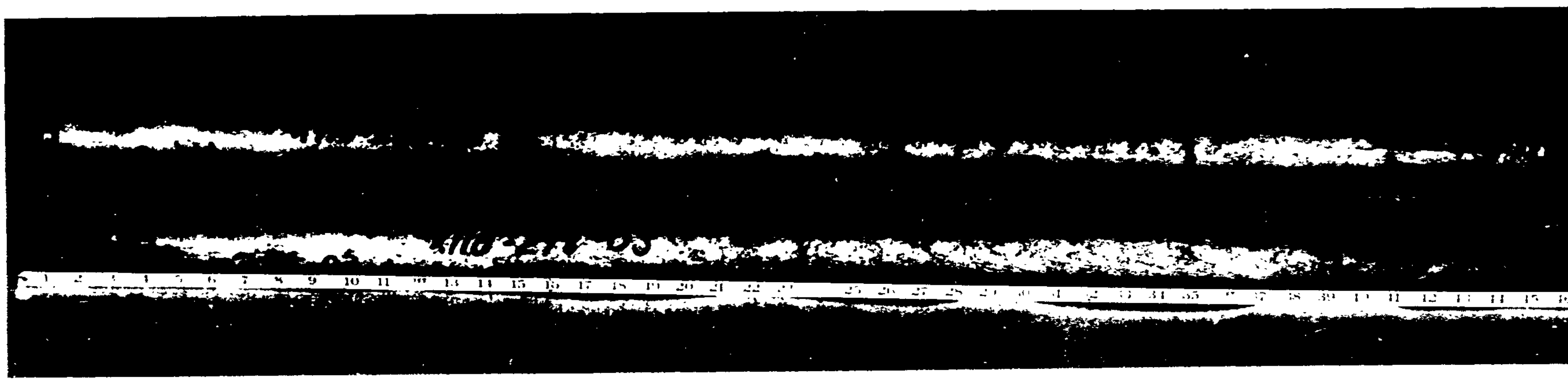
1) The glass on the barrel of the billet must be relatively low melting to effect a smooth extruded surface. The D-31 "T" extrusion development program had shown that use of a relatively high-melting glass caused a surface defect called glass "rub-in", or the impingement of too-hard glass particles into the extruding metal.

2) The glass in the bore of the billet must be higher melting than that applied to the barrel because the billet will be hotter at the bore (less radiation loss in transfer).

In the "Second Series", the first of this period, a low melting glass was applied to the barrel of the heated billets; two different high melting glasses were tried separately in the bore. Both the inside and outside surfaces of the tube blanks exhibited

tears and checks. The defects were attributed to poor lubrication. Two X-110 extrusions are shown in Figure 2. Figure 3 is a closeup view of the outside surfaces. The overall view of the three B-66 extrusions is shown in Figure 4, and a closeup view of two of them in Figure 5. The final extrusion in this series was made using a high melting glass on the barrel in contrast to the low melting glasses used previously. The outside surface was markedly improved. The extrusion B-66-277-01, is shown in Figures 4 and 5. The improvement is believed due to the enhanced lubrication provided by the higher melting glass.

The "Third Series" in Table 2 was the final and more extensive extrusion campaign. The damaged liner was replaced prior to this series so that cone type dies could be used in conjunction with the development of glass practice. The use of a high melting glass to lubricate both the inside and outside surfaces of the tube blank was continued from the previous series. The combination of cone die and high melting glass produced an acceptably smooth outside surface on the initial two extrusions but the inside surface exhibited rather deep indentations or tears transverse to the extrusion direction. The latter were significantly reduced but not entirely eliminated by using a low melting glass in the bore of the billets. This result was in contrast to the original premise above regarding I.D. glass lubrication, i.e., use a higher melting on the bore because of its higher temperature. A possible explanation for the high melting glass being the cause of the observed inside surface defects is as follows: the glass is chilled by the relatively cold mandrel, thereby decreasing its lubricity and also causing



<u>FIGURE 2</u>

X-110 TUBE BLANK EXTRUSIONS, SECOND SERIES

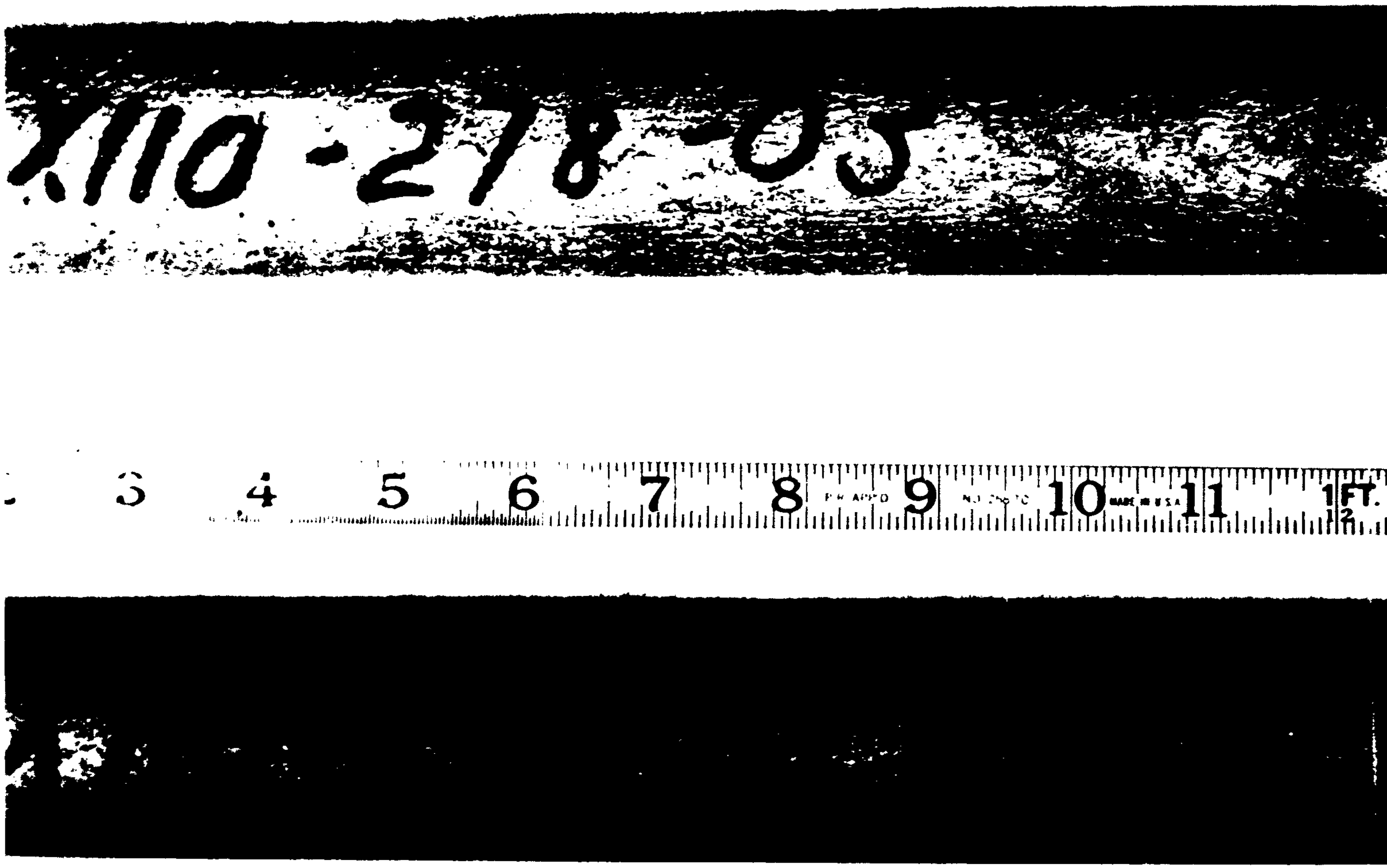


FIGURE 3

SANDBLASTED SURFACES OF X-110 TUBE BLANK EXTRUSIONS, SECOND SERIES

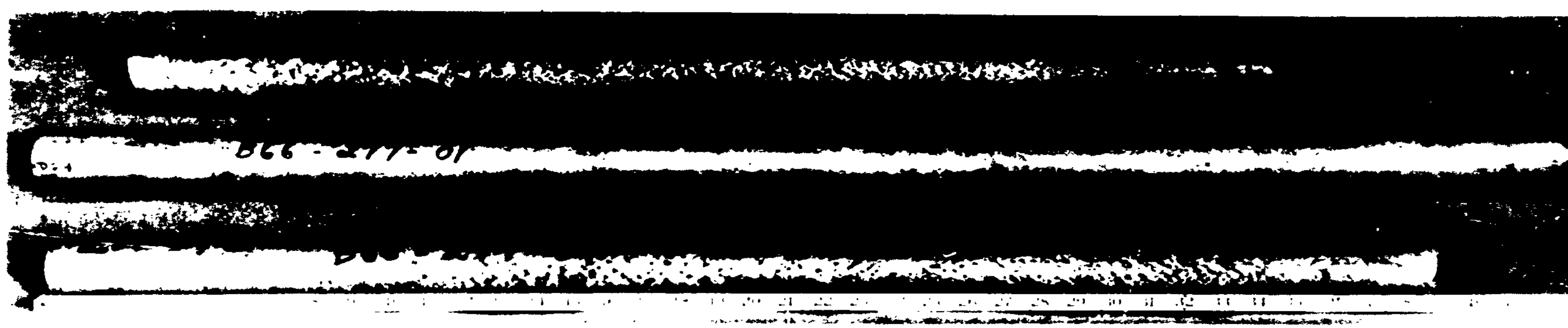


FIGURE 4

B-66 TUBE BLANK EXTRUSIONS, SECOND SERIES

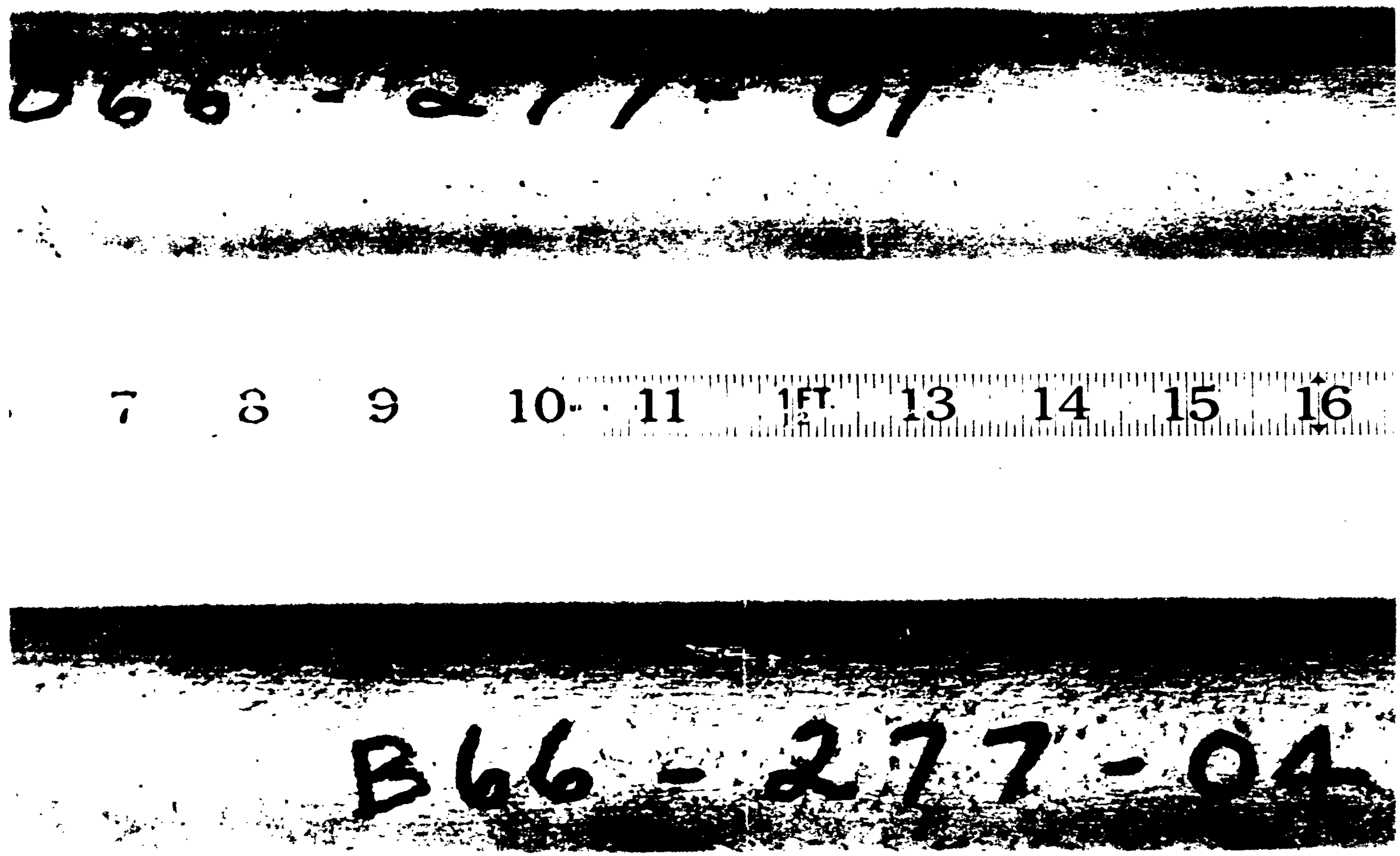


FIGURE 5

SANDBLASTED SURFACES OF B-66 TUBE BLANK EXTRUSIONS, SECOND SERIES

it to indent the extruding metal. Use of a lower melting glass will tend to decrease these undesirable effects.

The six B-66 extrusions of this series are shown in Figure 6. The degree of surface smoothness obtained is shown in the closeup view, Figure 7. The six X-110 extrusions appear in Figure 8, and in closeup, Figure 9. The extrusions were as-sandblasted.

One extrusion in the final series was made through a flat die using the same glass practice which, combined with the cone die, produced acceptable outside surfaces. A high melting glass cloth pad was placed against the flat die to effect a cone during extrusion. The outside surface of this extrusion - the X-110-280-01 blank in Figure 8 - exhibited light tears. The flat die extrusion surface (outside) was inferior to all of the cone die extrusion surfaces produced in this series.

In the two extrusion series a process was developed which produced both B-66 and X-110 tube blanks with nearly defect-free outside surfaces. The inside surfaces still exhibit transverse checks which are associated with improper glass lubrication. The essential features of this process are -

1) extrusion ratio of approximately 8:1
2) billet temperature in the range 3000-3200°F.; transfer time under 35 seconds
3) 90° included-angle cone die, ZrO_2 coated; ZrO_2 coated mandrel
4) high melting glass applied to the barrel or outside surface of the billet
5) low melting glass applied to the bore of the billet

FIGURE 6

B-66 TUBE BLANK EXTRUSIONS, THIRD SERIES

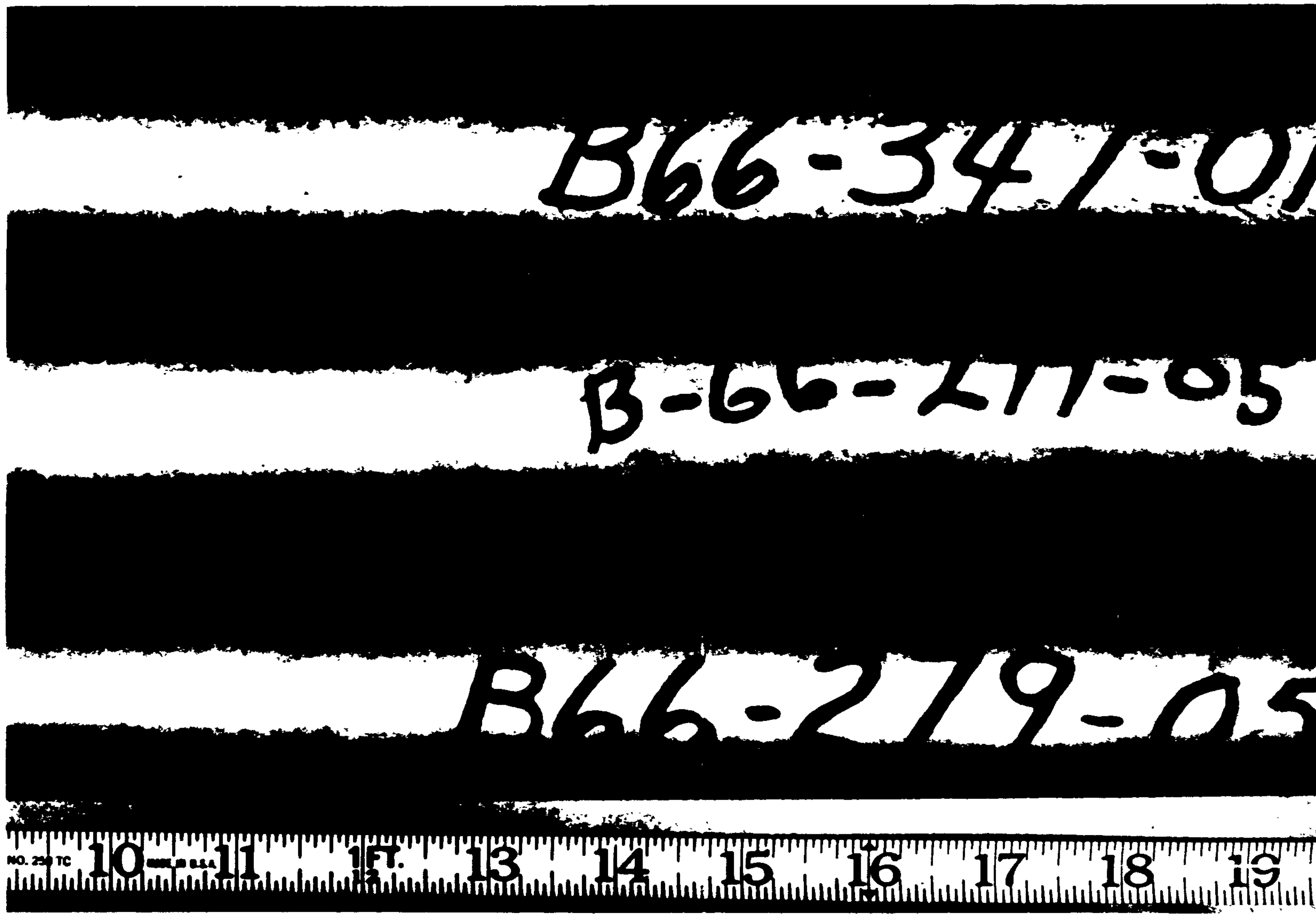


FIGURE 7

SANDBLASTED SURFACES OF B-66 TUBE BLANK EXTRUSIONS, THIRD SERIES

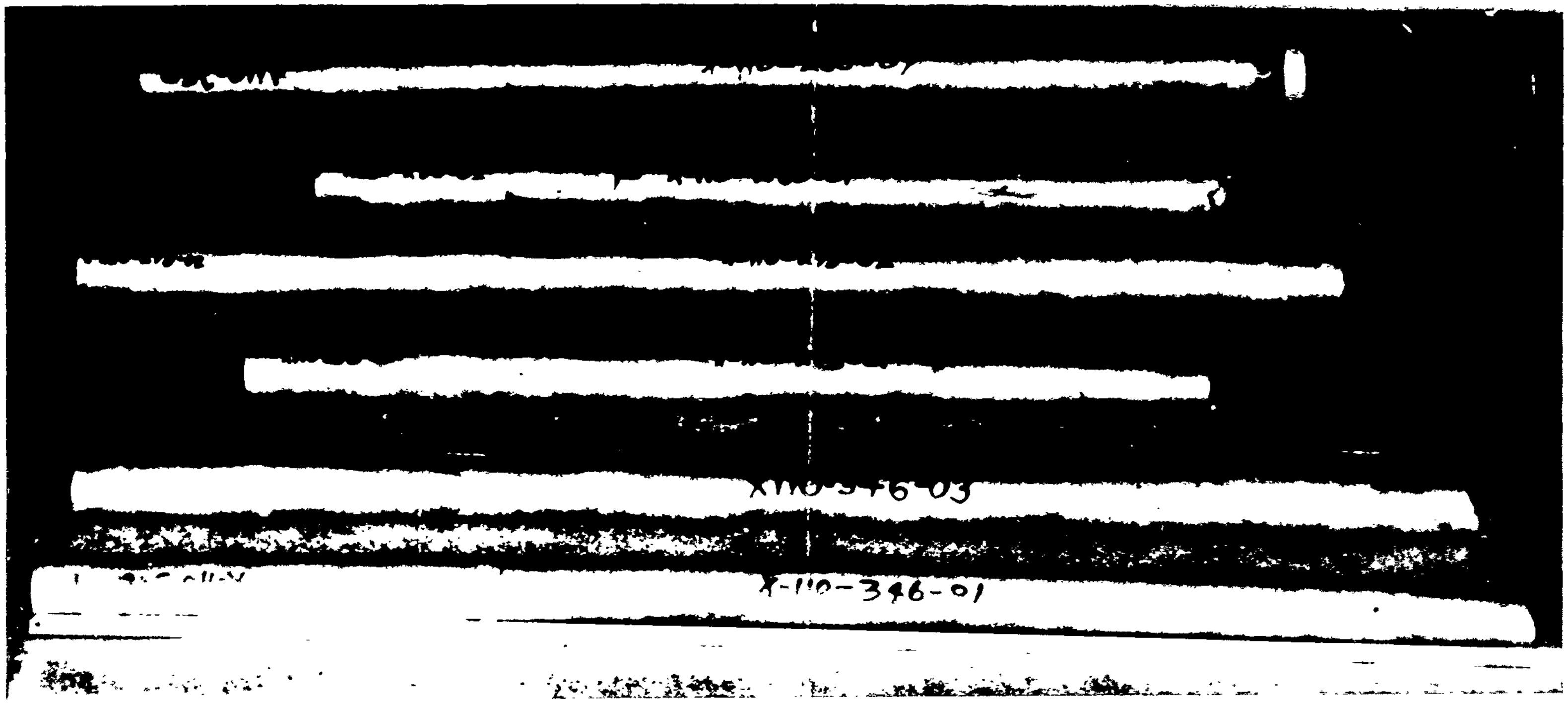


FIGURE 8

X-110 TUBE BLANK EXTRUSIONS, THIRD SERIES

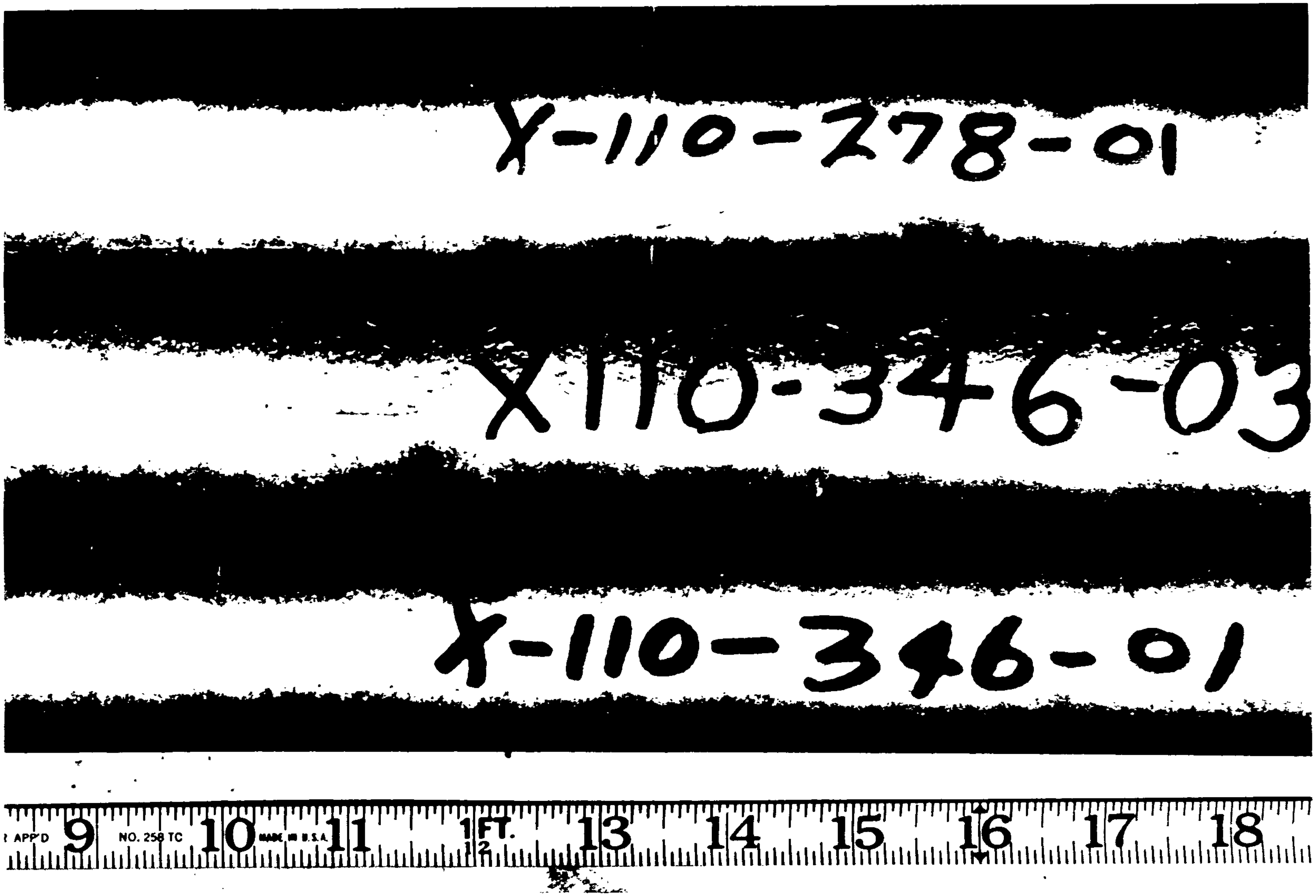


FIGURE 9

SANDBLASTED SURFACES OF X-110 TUBE BLANK EXTRUSIONS, THIRD SERIES

C. METALLOGRAPHY AND TESTING

1. Chemical analysis

Selected tube blanks were sampled and analyzed. The results are presented in Table 3. Both B-66-347-01 and X-110-346-01 were produced from the additional ingots melted in this reporting period.

TABLE 3

CHEMICAL ANALYSIS OF AS-EXTRUDED B-66 AND X-110 TUBE BLANKS

Tube Blank	Element							
	O(ppm)	H(ppm)	N(ppm)	C(ppm)	W(%)	Zr(%)	Mo(%)	V(%)
B-66-277-05	125	14	56	126	-	.88	4.9	5.4
B-66-347-01	125	24	63	128	-	.93	4.8	5.3
B-66 Specification[1]	300 max	N.R.	200 max	200 max	-	.85-1.3	4.5-5.5	4.5-5.5
X-110-278-02	48	29	35	1060	9.6	.90	-	-
X-110-346-01	31	4	52	1180	9.6	.89	-	-
X-110 Specification[2]	400 max	20 max	100 max	800-1200	9.0-11.0	0.75-1.25	-	-

1. Reference: Westinghouse Electric Corporation, "Special Technical Data" bulletin 52-364, p. 1.

2. Reference: Tentative Du Pont Product Specification DPC (P)-1101.

2. Metallography

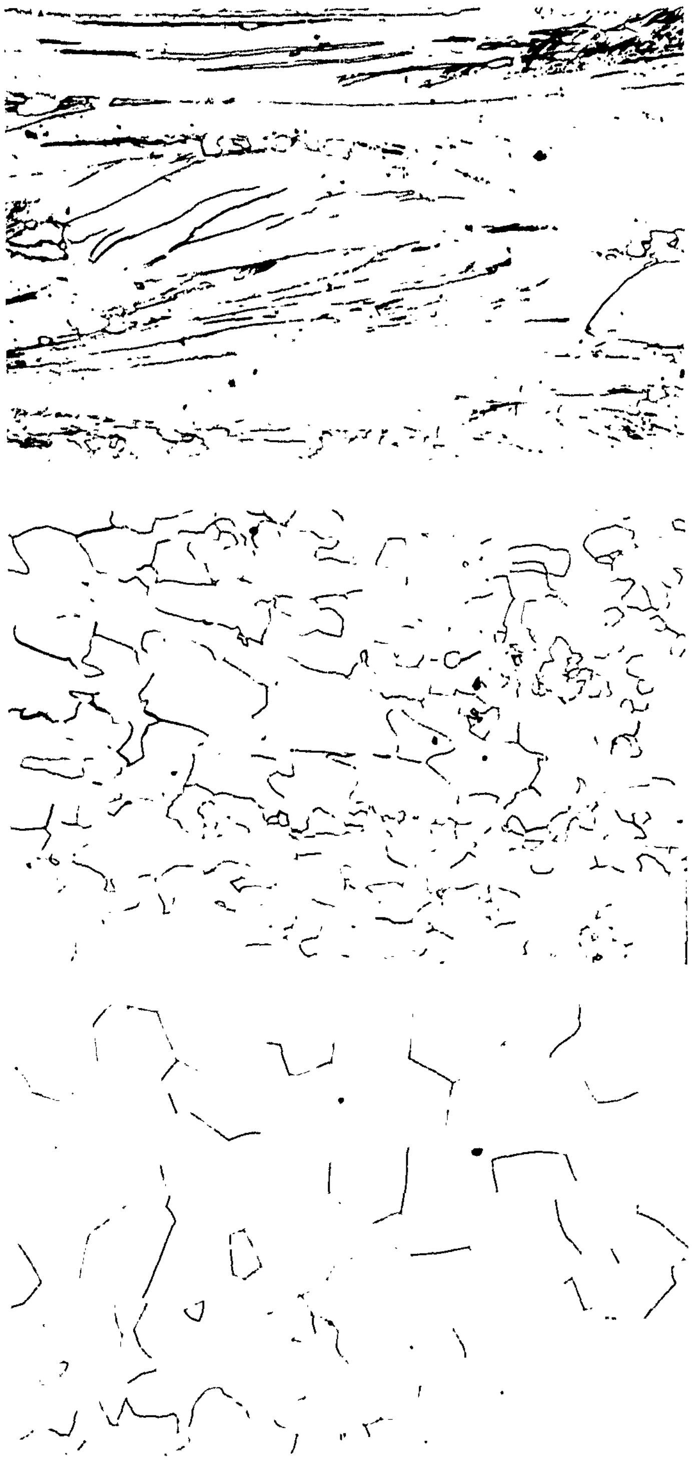
Samples from the front and back of selected tube blanks from the "Third Series" of extrusions were examined for microstructure and hardness in the as-extruded and annealed conditions. The results are summarized in Table 4. The microstructure (150X) of B-66 (347-01, back) and X-110 (346-01, back) are shown in Figures 10 and 11 respectively. Some recrystallization occurred in the B-66 at 2200°F/1 hr., with complete recrystallization and grain growth being evident after 2600°F/2 hrs. Similar annealing response was reported (1) for B-66 from an earlier tube blank extrusion in this program.

The X-110 microstructures reflect the lower extrusion temperatures - approximately 200°F. - employed in the present series as compared with the initial series (1). No recrystallization is evident, as compared with 5-80% present in the initial extrusions. Moreover, no change in microstructure was observed on annealing the initial extrusion samples at 2600°F/1 hr., whereas the present samples, Table 4, exhibited partial recrystallization after 2600°F/2 hrs. This response can also be attributed to a lower extrusion temperature.

TABLE 4

MICROSTRUCTURE AND HARDNESS OF B-66 AND X-110 TUBE BLANKS

Tube Blank	Microstructure and Hardness in Condition -		
	As-Extruded	$2200^{o}F$/1 hr.	$2600^{o}F$/2 hrs.
I. B-66:			
277-05 front	cold-worked; incip. recryst.; R_B 98	10-60% recryst.; R_B 94	100% recryst.; ASTM 3-6 R_B 93
277-05 back	10-40% recryst.; R_B 95	>90% recryst.; ASTM 4-8; R_B 92	100% recryst.; ASTM 3-7; R_B 92
347-01 front	cold worked; incip. recryst.; R_B 102	50-80% recryst.; R_B 92	100% recryst.; ASTM 2-7; R_B 92
347-01 back	10-25% recryst.; R_B 97	50-100% recryst.; R_B 90	100% recryst.; ASTM 3-7; R_B 84
II. X-110:			
278-02 front	cold worked; R_B 93	cold worked; R_B 88	<20% recryst.; R_B 84
278-02 back	cold worked; R_B 92	cold worked; R_B 89	10-20% recryst.; R_B 82
346-01 front	cold worked; R_B 91	cold worked; R_B 88	10-20% recryst.; R_B 83
346-01 back	cold worked; R_B 92	cold worked; R_B 93	25-50% recryst.; R_B 78



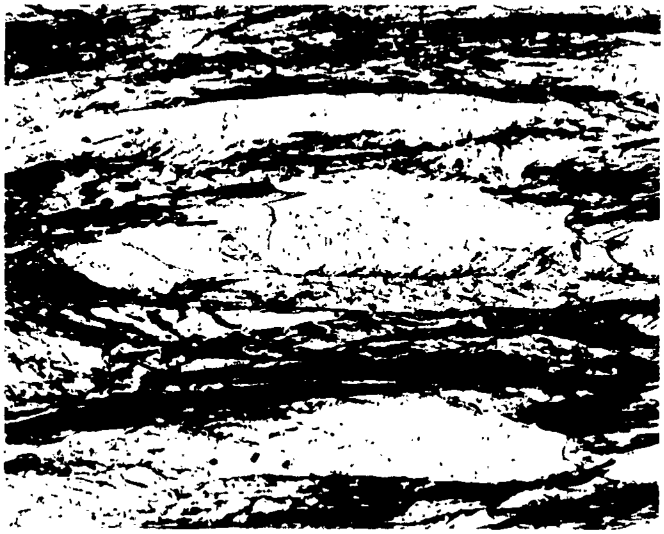
A. As-Extruded

B. 2200°F/1 hr.

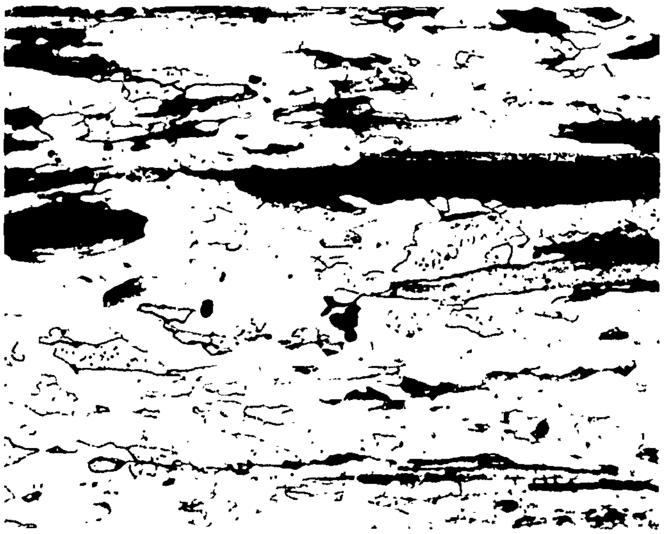
C. 2600°F/2 hrs.

FIGURE 10

MICROSTRUCTURES OF B-66-347-01 TUBE BLANKS, (BACK)
BOTH AS-EXTRUDED AND AFTER ANNEALING. 150X.



A. As-Extruded



B. 2600°F/2 hrs.

FIGURE 11

MICROSTRUCTURES OF X-110-346-01 TUBE BLANKS (BACK), BOTH AS-EXTRUDED AND AFTER ANNEALING. 150X.

3. Tensile Tests, Room Temperature

Longitudinal tensile specimens were prepared from the back end of both a B-66 (347-01) and an X-110 (278-02) tube blank. The specimen size was 0.125" diameter x .500" gage length. Duplicate specimens were tested in three conditions - as-extruded, annealed 2200°F/1 hr., and annealed 2600°F/2 hrs. These conditions correspond to the metallographic samples described above. The tensile test results are present in Table 5. The strain rate was controlled at 0.005"/"/min. to yield, then 0.05"/"/min. to failure.

Both the B-66 and the X-110 samples exhibited tensile elongations of the order of 20% in the as-extruded condition. Annealing of the B-66 for 2200°F/1 hr., which resulted in almost complete recrystallization (Table 5), increased the % reduction in area significantly. The samples of B-66 annealed at 2600°F/ 2 hr. (completely recrystallized, with considerable grain growth, Table 4) did not have improved elongation and reduction-in-area values over the material annealed at 2200°F/1 hr.

Annealing of the X-110 at 2200°F/1 hr. resulted in a decrease in yield strength but a slight increase in ultimate strength. The 2600°F. anneal, which produced 10-20% recrystallization, reduced the strength values considerably but did not increase the elongation or reduction-in-area values significantly.

TABLE 5

TENSILE TESTS (ROOM TEMPERATURE) OF SAMPLES FROM B-66 AND X-110 TUBE BLANKS[1]

Code	Condition	U.T.S.,ksi	0.2% Y.S.,ksi	% E in 4D	% RA	Hardness, R_B
B-66-347-01	As-extruded	109.2	84.5	23	45	96-98
		109.6	86.3	28	51	
	2200°F/1 hr.	98.5	77.3	30	71	90
		100.8	78.9	30	79	
	2600°F/2 hrs.	93.8	74.3	30	72	82-86
		94.6	75.7	34	70	
X-110-278-02	As-extruded	94.0	78.0	18	67	91-93
		93.2	76.8	17	56	
	2200°F/1 hr.	96.0	69.9	21	63	89
		96.7	70.1	28	65	
	2600°F/2 hrs.	78.5	59.1	24	62	81-82
		80.1	59.5	31	64	

1 Longitudinal samples taken from back of tube blanks.

D. TUBE BLANK CONDITIONING AND ANNEALING

The sizes of the extrusion dies and mandrels were selected to allow for approximately 0.050" "cleanup" on the inside and outside diameters of the tube blanks to the starting sizes recommended by Wolverine Tube. The tentative process for conditioning and annealing was as follows:

1. sandblast
2. straighten
3. condition inside surface by honing
4. condition outside surface by machining or centerless grinding
5. anneal
6. inspect

The straightening step has not been successful despite repeated trials. B-66 and X-110 tube blanks were fractured on both a roller straightener and a gag press. The brittle behavior at ambient temperature is believed to be caused by the layer of contamination from the high temperature extrusion, combined with the numerous surface defects. The purpose of straightening, of course, is to permit the machining or grinding away of the contaminated surface. For the final straightening trial, a batch of tube blanks was first conditioned on the inside surface by honing (straightness not required). After honing, the surfaces still contained some extrusion defects. To remove these defects completely at this stage of processing would have required excessive metal removal. Next the tube blanks were deep pickled on inside and out, approximately 0.010" of stock being removed

from the diameters (check on O.D. only). The metallographic study had shown that the depth of contamination in the as-extruded state was up to .010" deep. The first blank selected for roller straightening had a total runout of 1/8-1/4" in three feet. The tube blank was given about ten straightening passes at ambient temperature during which time it became too hot to handle without gloves. No fractures were visible. The degree of straightness was 0.025" TIR, not quite adequate for centerless grinding. At the very beginning of the next pass the blank fractured, Figure 12. The fracture closeup appears in Figure 13. The fracture probably occurred by crushing of the lead end between the rolls. The remainder of the blanks in this batch were hand conditioned by disk sander to blend in extrusion defects then deep pickled once again to reduce the outside diameter below the maximum starting size - 1.750" diameter - of the Wolverine tube reducer.

The initial batch of tube blanks produced in this phase of the contract were conditioned on the outside surface by machining or belt sanding. Straightness was not a problem because the blanks were only 1'-2' long. The shorter lengths were the result of 1) breakage in straightening trials and 2) extensive cropping to remove serious extrusion defects. Defects still in the surface after machining the O.D. to about 1.750" were removed by hand grinding. The inside surfaces were conditioned by honing to remove a majority of the extrusion defects. The inside surfaces were not entirely conditioned for this would have resulted in excessive metal loss.

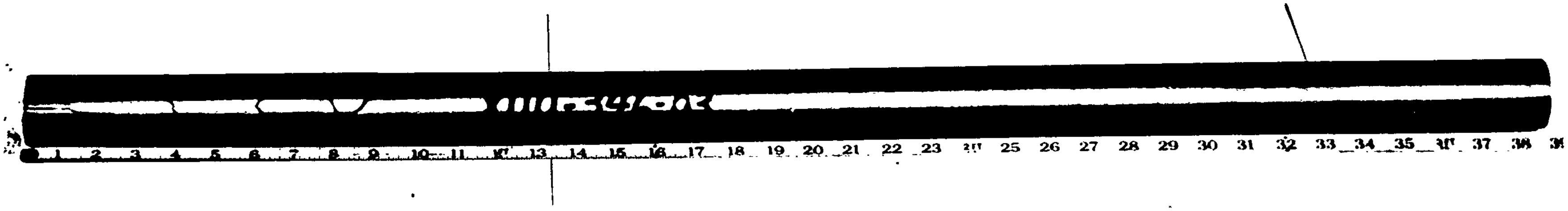


FIGURE 12

X-110 TUBE BLANK FRACTURED IN ROLLER STRAIGHTENING TRIAL

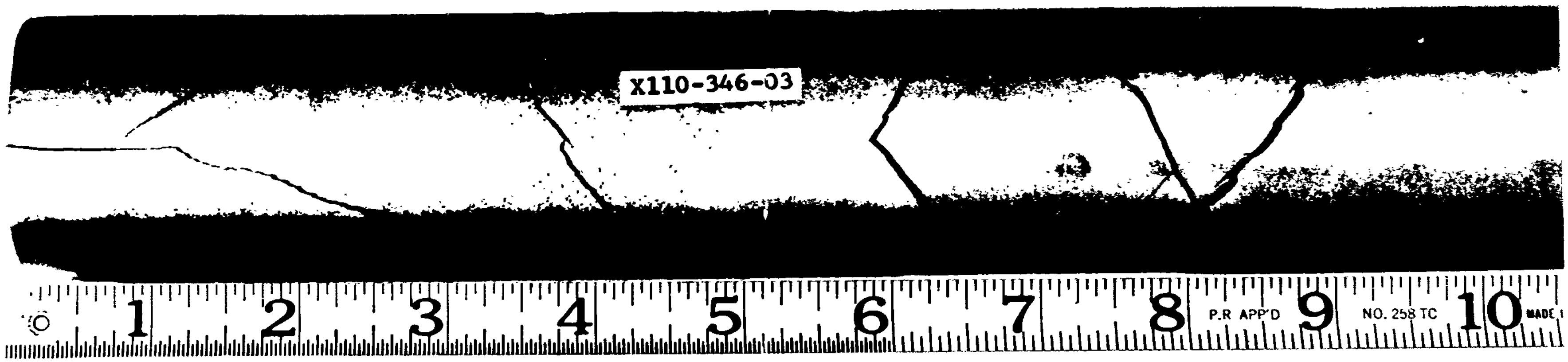


FIGURE 13

CLOSEUP OF X-110 TUBE BLANK FRACTURED IN ROLLER STRAIGHTENING TRIAL

All tube blanks were annealed at 2550-2600°F/1 hr. in the large sheet vacuum furnace at the Du Pont Metals Center prior to the first tube reduction.

E. TUBE REDUCTION

Wolverine Tube, Allen Park, Michigan, is the subcontractor who will perform the tube reduction in a cooperative program with Du Pont. Wolverine recommended one process schedule for producing the 1/2" and 3/8" O.D. tubes and a second schedule for producing the 1/4" O.D. tubes. These processing schedules were agreed upon by Du Pont. They are reproduced below as Schedule No. 1 (1/2" and 3/8" O.D.) and Schedule No. 2 (1/4" O.D.). Tentatively two tube reduction passes are planned between intermediate anneals after the first tube reduction-anneal step has been completed. The reduction-anneal cycle will be evaluated as the program progresses.

The first batch of tube blanks, consisting of ten pieces, was inspected by Wolverine. They performed further hand conditioning on the outside surfaces of certain pieces to remove what were considered objectionable defects. Following this the pieces were deep pickled and carefully inspected. Conditioned pieces are shown in Figure 14. Local grinding marks are visible in the top two pieces. The Wolverine personnel carefully recorded in their inspection reports the location of discontinuities on the inside and outside surfaces. Reinspection of these locations following the first tube reduction would indicate what degree of local conditioning of the starting blanks is tolerable.

PROCESS SCHEDULE NO. 1

Tube Reduction to 1/2" and 3/8" O.D. Tubing

Finish target sizes: .500" ± .005" O.D. x .060" ± 10% wall (40%)[1]

.375" ± .005" O.D. x .060" ± 10% wall (40%)[1]

Starting material: 1.750" O.D. x .300" wall

	Operation	Size	% Wall Reduction	% Area Reduction
1.	Inspect	1.750" ODx.300" wall	-	-
2.	Tube reduce	1.375" ODx.210" wall	30	43.8
3.	Inspect			
4.	Anneal			
5.	Salvage; inspect			
6.	Tube reduce	1.062" ODx.147" wall	30	44.9
7.	Inspect			
8.	Salvage; inspect			
9.	Tube reduce	.812" ODx.103" wall	30	45.9
10.	Inspect			
11.	Anneal			
12.	Salvage; inspect			
13.	Tube reduce	.625" ODx.073" wall	30	45.0
14.	Inspect			
15.	Salvage; inspect			
16.	Tube reduce			
	half to	.500" ODx.060" wall	30	34.1
	half to	.375" ODx.060" wall	30	53.2
17.	Inspect			
18.	Anneal			
19.	Salvage			
20.	Final inspection			

[1] Percentage based on total material supplied by Du Pont

PROCESS SCHEDULE NO. 2

Tube Reduction to 1/4" O.D. Tubing

Finish target size: .250" ± .005" O.D. x .015/.020" wall (20%)[1]

Starting material: 1.750" O.D. x .250" wall

	Operation	Size	% Wall Reduction	% Area Reduction
1.	Inspect	1.750" ODx.250" wall	-	-
2.	Tube reduce	1.375" ODx.160" wall	35	47.5
3.	Inspect			
4.	Anneal			
5.	Salvage; inspect			
6.	Tube reduce	1.062" ODx.100" wall	35	50.8
7.	Inspect			
8.	Salvage; inspect			
9.	Tube reduce	.812" ODx.065" wall	35	49.5
10.	Inspect			
11.	Anneal			
12.	Salvage; inspect			
13.	Tube reduce	.625" ODx.038" wall	42	53.9
14.	Inspect			
15.	Salvage; inspect			
16.	Tube reduce	.500" ODx.022" wall	42	52.8
17.	Inspect			
18.	Anneal			
19.	Salvage; inspect			
20.	Tube reduce	.250" ODx.018" wall	18	60.6
21.	Inspect			
22.	Anneal			
23.	Salvage			
24.	Final inspection			

1 Percentage based on total material supplied by Du Pont

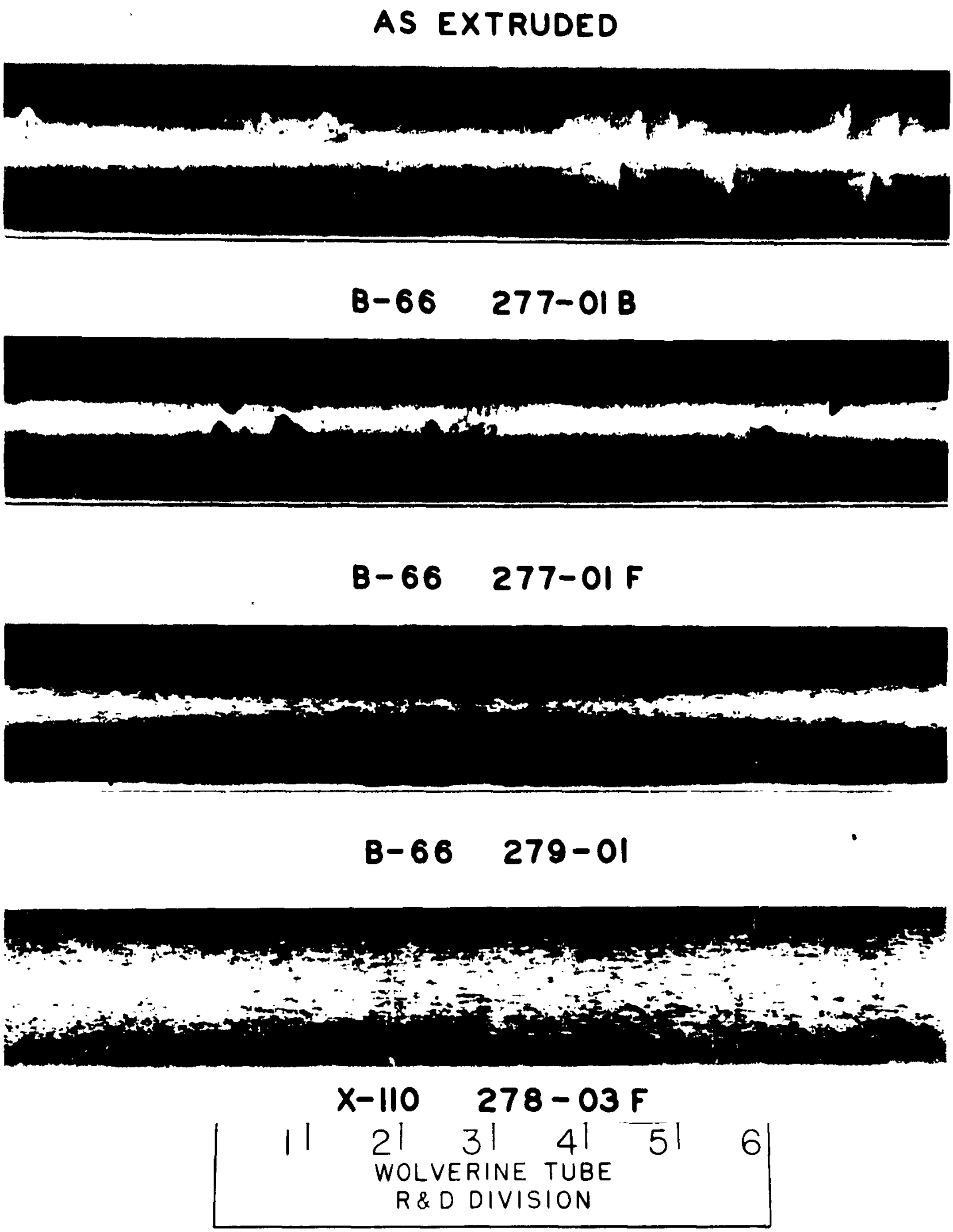


FIGURE 14

CONDITIONED TUBE BLANKS PRIOR TO FIRST TUBE REDUCING PASS

The first tube reduction of the first batch of ten pieces was made in January, 1963, at Wolverine. The X-110 pieces reduced satisfactorily. The outside surfaces were fair to good. Defects still exist on the inside surfaces; they have been recorded on the Wolverine inspection reports following the tube reduction. An overall view of the tube reduced pieces is shown in Figure 15. The key notches in one end of the pieces were used to attach the pieces to a holder during tube reducing. Figure 16 is a closeup of the tube-reduced surfaces.

The first B-66 tube blank tried in tube reducing (ambient temperature) cracked radially, Figure 17. Close examination of this piece under low power magnification revealed small residual extrusion defects which could have caused cracking. Two other B-66 tube blanks were subsequently deep pickled on the inside and outside surfaces then carefully hand-conditioned once again by Wolverine personnel. These pieces also cracked radially in tube reducing. They are shown in Figures 15 and 16 (B-66-277-01F and -01B). The cracking is believed due to the residual extrusion defects which exist on the inside - and perhaps the outside - surface of the B-66 pieces. The behavior of this alloy is in contrast to the X-110 which tube reduced satisfactorily despite the presence of similar surface defects.

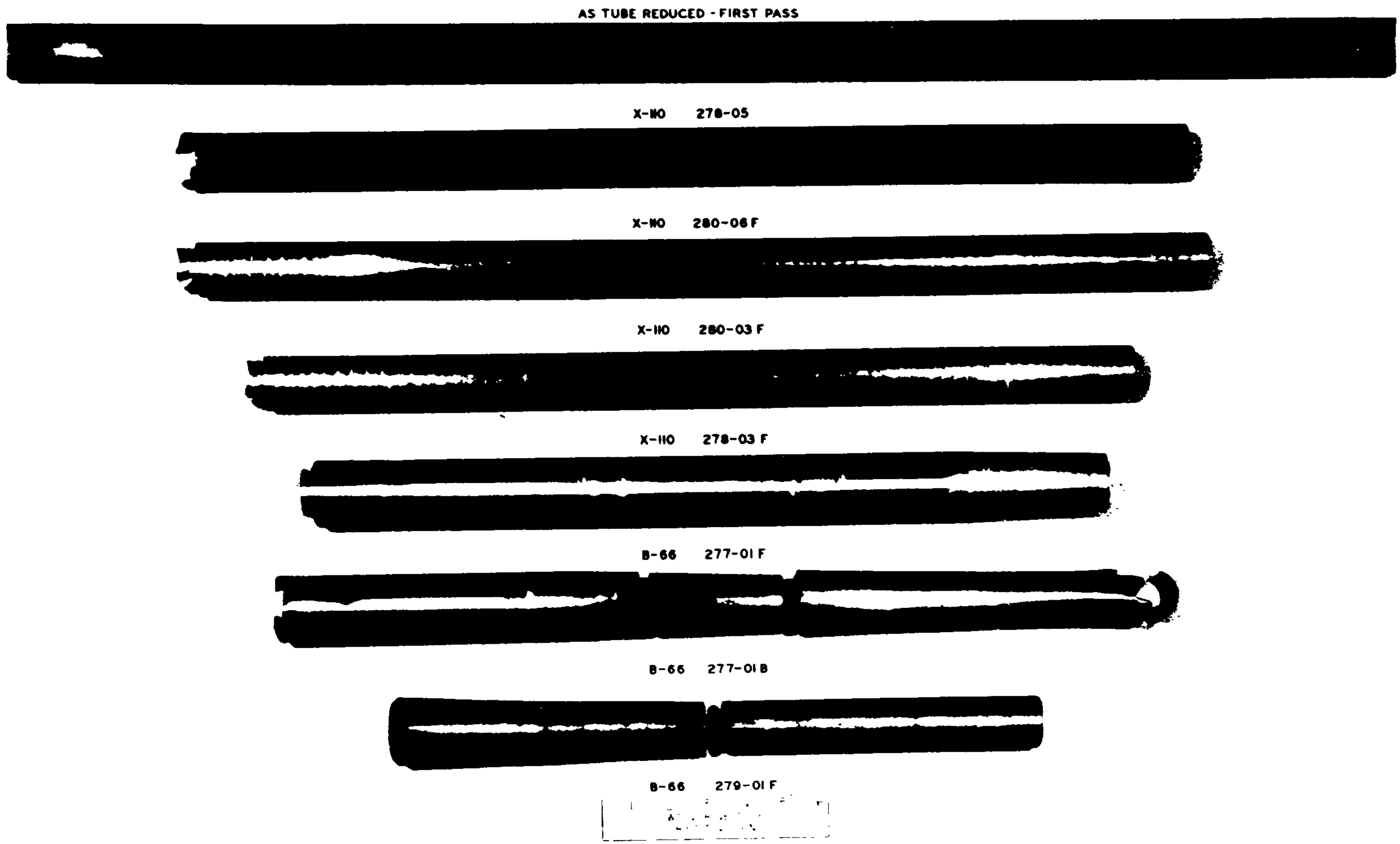


FIGURE 15

TUBE BLANKS AFTER FIRST TUBE REDUCING PASS

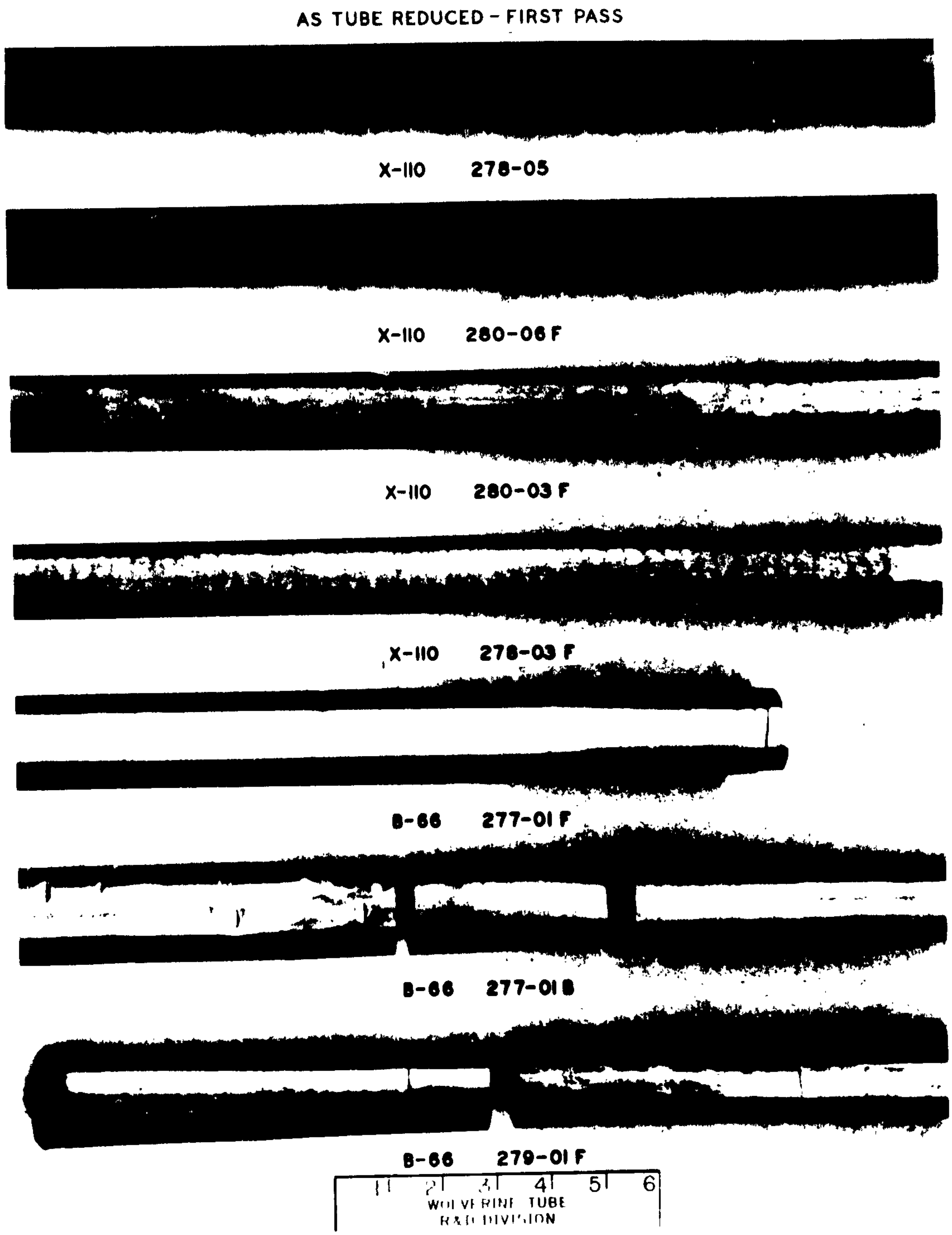


FIGURE 16

SURFACE OF TUBE BLANKS AFTER FIRST TUBE REDUCING PASS

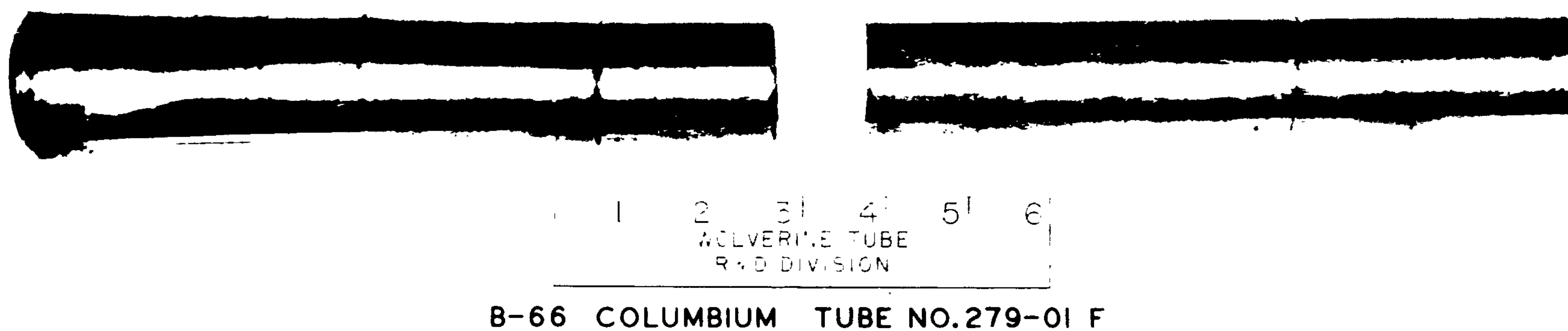


FIGURE 17

B-66 TUBE BLANK WHICH CRACKED IN FIRST TUBE REDUCING PASS

IV. FUTURE PROGRAM

A. TUBE BLANK EXTRUSION

Three additional tube blank extrusions - all B-66 - will be made early in February, 1963. With the completion of these extrusions, all of the material in the program (except one solid billet each of B-66 and X-110) will have been converted into tube blanks. In this series an attempt will be made to eliminate the extrusion defects on the internal surface which have persisted throughout the extrusion development program.

B. TUBE REDUCTION

The first batch of X-110 tube blanks, satisfactorily tube reduced through the first pass, will be annealed and conditioned prior to further tube reducing (see Processing Schedules, pps. 37 and 38). The second batch of X-110 tube blanks, presently conditioned at the extruded size, will be annealed and delivered to Wolverine for the initial tube reduction. The in-process microstructure will be examined.

The cracking problem with the B-66 tube blanks must be resolved. The following steps will be taken:

1. Tube reduce an unannealed blank (as-extruded). The three tubes tried were in the recrystallized condition. Although the tensile data reported in Table 5 for B-66 does not indicate a decrease in ductility on annealing, the fracture might be associated with recrystallized grains. The B-66 fractures progressed both around (inter-) and through (trans-) grains.

2. Tube reduce a blank which has been machined then deep pickled <u>all over</u> to virtually eliminate the possibility of a fracture initiating at a residual extrusion defect.
3. If steps 1) and 2) still produce cracking on tube reducing, adjust the first pass to a lighter reduction. If the piece reduces without cracking, perform an anneal.
4. Determine the bend transition temperature for as-extruded and annealed material. The results might indicate that cold tube reducing of B-66 is not feasible. Warm tube reducing, however, is not considered to be within the scope of this contract.

V. ACKNOWLEDGEMENTS

The author wishes to acknowledge the contributions to this program by A. W. Dana, Jr., for technical supervision, R. W. Felber for extrusion operations supervision, and J. A. Crane for metallography and mechanical testing.

Wolverine Tube is conducting the tube reduction portion of this program. The valuable contributions of personnel of that company are acknowledged, in particular those of J. C. Huber, the engineer in charge, F. C. Eddens, Manager, Special Metals Department, and L. B. Moorman, Project Metallurgist.

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January, 1963

ASD Project No. 7-775 (VII)
(Contract AF33(600)-40700)
Unclassified Report

The development of a tube blank extrusion process for B-66 (Cb-5Mo-5V-1Zr) and X-110 (Cb-10W-1Zr-.1C) is described. The tube reducing program
(over)

UNCLASSIFIED

1. Extrusion of columbium
2. Physical metallurgy
3. Mechanical metallurgy
4. Primary breakdown
5. Tube Blank Extrusion

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Metal Products
Pigments Department
Wilmington, Delaware
COLUMBIUM AND COLUMBIUM ALLOY EXTRUSION PROGRAM
January, 1963

ASD Project No. 7-775 (VII)
(Contract AF33(600)-40700)
Unclassified Report

The development of a tube blank extrusion process for B-66 (Cb-5Mo-5V-1Zr) and X-110 (Cb-10W-1Zr-.1C) is described. The tube reducing program
(over)

UNCLASSIFIED

1. Extrusion of columbium
2. Physical metallurgy
3. Mechanical metallurgy
4. Primary breakdown
5. Tube Blank Extrusion

E. I. du Pont de Nemours & Co., Inc.
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